

Analytical Laboratory

13339 Hagers Ferry Road Huntersville, NC 28078-7929 McGuire Nuclear Complex - MG03A2 Phone: 980-875-5245 Fax: 980-875-4349

Order Summary Report

Order Number:	J11100039							
Customer Name(s):	Bill Kennedy, Melonie Martin, Wayne	e Chapman,	Tom Johnson					
Customer Address:	3195 Pine Hall Rd							
	Mailcode: Belews Steam Station							
	Belews Creek, NC 28012							
Lab Contact:	Jason C Perkins	Phone:	980-875-5348					
Report Authorized By: (Signature)		Date	9 :	10/27/2011				

Program Comments:

Please contact the Program Manager (Jason C Perkins) with any questions regarding this report.

Data Flags & Calculations:

Any analytical tests or individual analytes within a test flagged with a Qualifier indicate a deviation from the method quality system or quality control requirement. The qualifier description is found at the end of the Certificate of Analysis (sample results) under the qualifiers heading. All results are reported on a dry weight basis unless otherwise noted.

Data Package:

This data package includes analytical results that are applicable only to the samples described in this narrative. An estimation of the uncertainty of measurement for the results in the report is available upon request. This report shall not be reproduced, except in full, without the written consent of the Analytical Laboratory. Please contact the Analytical laboratory with any questions. The order of individual sections within this report is as follows:

Job Summary Report, Sample Identification, Technical Validation of Data Package, Analytical Laboratory Certificate of Analysis, Analytical Laboratory QC Reports, Sub-contracted Laboratory Results, Customer Specific Data Sheets, Reports & Documentation, Customer Database Entries, Test Case Narratives, Chain of Custody (COC)

Certification:

The Analytical Laboratory holds the following State Certifications: North Carolina (DENR) Certificate #248, South Carolina (DHEC) Laboratory ID # 99005. Contact the Analytical Laboratory for definitive information about the certification status of specific methods.

Sample ID's & Descriptions:

Page 2 of 27

Sample ID	Plant/Station	Collection Date and Time	Collected By	Sample Description
2011021606	BELEWS	12-Oct-11 11:08 AM	ТО	FGD Purge Eff
2011021607	BELEWS	12-Oct-11 10:56 AM	ТО	EQ TANK EFF.
2011021608	BELEWS	12-Oct-11 10:29 AM	ТО	BIOREACTOR 1 INF.
2011021609	BELEWS	12-Oct-11 10:35 AM	ТО	BIOREACTOR 2 INF.
2011021610	BELEWS	12-Oct-11 10:41 AM	ТО	BIOREACTOR 2 EFF.
2011021611	BELEWS	12-Oct-11 10:22 AM	ТО	FILTER BLANK
2011021612	BELEWS	12-Oct-11 10:22 AM	ТО	Trip Blank
2011021613	BELEWS	12-Oct-11 10:29 AM	ТО	BIOREACTOR 1 INF.
2011021614	BELEWS	12-Oct-11 10:29 AM	ТО	HG BLANK BIOREACTOR 1 INF.
2011021615	BELEWS	12-Oct-11 10:35 AM	ТО	BIOREACTOR 2 INF.
2011021616	BELEWS	12-Oct-11 10:35 AM	ТО	Hg Blk BioReactor 2 Inf
2011021617	BELEWS	12-Oct-11 10:41 AM	ТО	BIOREACTOR 2 EFF.
2011021618	BELEWS	12-Oct-11 10:41 AM	ТО	Hg Blk BioReactor 2 Eff

Checklist:

Reviewed By:

DataBase Administrator

COC and .pdf report are in agreement with sample and analyses (compliance programs and procedure		✓ Yes	☐ No
All Results are less than the laboratory reporting lin	nits.	Yes	✓ No
All laboratory QA/QC requirements are acceptable.		✓ Yes	☐ No
The Vendor Laboratories have been qualified by the Analytical Laboratory	е	Yes	
Report Sections Included:			
✓ Job Summary Report	✓ Sub-contr	acted Laborato	ory Results
✓ Sample Identification	☐ Customer	Specific Data	Sheets, Reports, & Documentation
✓ Technical Validation of Data Package	☐ Customer	Database Ent	ries
✓ Analytical Laboratory Certificate of Analysis	✓ Chain of 0	Custody	
☐ Analytical Laboratory QC Report	✓ Electronic	: Data Delivera	able (EDD) Sent Separately

Date:

10/27/2011

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Order # J11100039

Site: FGD Purge Eff Sample #: 2011021606

Collection Date: 12-Oct-11 11:08 AM Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	Method	Analysis Date/Time	Analyst
INORGANIC IONS BY IC							
Bromide	88	mg/L		10	EPA 300.0	25-Oct-11 07:45	JAHERMA
MERCURY (COLD VAPOR)	IN WATER						
Mercury (Hg)	317	ug/L		5	EPA 245.1	21-Oct-11 09:04	AGIBBS
TOTAL RECOVERABLE ME	TALS BY ICP						
Boron (B)	182	mg/L		0.5	EPA 200.7	24-Oct-11 13:15	DJSULL1
DISSOLVED METALS BY IC	°D_MS						
Selenium (Se)	<u>7F-IVIS</u> 1130	ug/L		10	EPA 200.8	19-Oct-11 12:01	KRICHAR
,		· ·		10	2.7.200.0	10 000 11 12.01	14.4013.41
TOTAL RECOVERABLE ME	TALS BY ICP-MS						
Arsenic (As)	159	ug/L		10	EPA 200.8	19-Oct-11 12:25	KRICHAR
Chromium (Cr)	228	ug/L		10	EPA 200.8	19-Oct-11 12:25	KRICHAR
Copper (Cu)	126	ug/L		10	EPA 200.8	19-Oct-11 12:25	KRICHAR
Nickel (Ni)	188	ug/L		10	EPA 200.8	19-Oct-11 12:25	KRICHAR
Selenium (Se)	6080	ug/L		20	EPA 200.8	19-Oct-11 12:25	KRICHAR
Silver (Ag)	< 10	ug/L		10	EPA 200.8	19-Oct-11 12:25	KRICHAR
Zinc (Zn)	228	ug/L		20	EPA 200.8	19-Oct-11 12:25	KRICHAR
SELENIUM SPECIATION							
Vendor Parameter	Complet	е			V_AS&C		
TOTAL DISSOLVED SOLID	<u>S</u>						
TDS	18000	mg/L		200	SM2540C	19-Oct-11 13:25	TJA7067

Site: EQ TANK EFF. Sample #: 2011021607

Collection Date: 12-Oct-11 10:56 AM Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	Method	Analysis Date/Time	Analyst		
MERCURY (COLD VAPOR) IN WAT	<u>ER</u>								
Mercury (Hg)	199	ug/L		2.5	EPA 245.1	21-Oct-11 09:07	AGIBBS		
TOTAL RECOVERABLE METALS BY ICP									
Boron (B)	163	mg/L		0.5	EPA 200.7	24-Oct-11 13:19	DJSULL1		
DISSOLVED METALS BY ICP-MS									
Selenium (Se)	1150	ug/L		10	EPA 200.8	19-Oct-11 12:04	KRICHAR		

2011021607

Certificate of Laboratory Analysis

This report shall not be reproduced, except in full.

Order # J11100039

Site: EQ TANK EFF. Sample #:

Collection Date: 12-Oct-11 10:56 AM Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	Method	Analysis Date/Time	Analyst
TOTAL RECOVERABLE METALS	BY ICP-MS						
Arsenic (As)	113	ug/L		10	EPA 200.8	19-Oct-11 11:19	KRICHAR
Chromium (Cr)	168	ug/L		10	EPA 200.8	19-Oct-11 11:19	KRICHAR
Copper (Cu)	90.2	ug/L		10	EPA 200.8	19-Oct-11 11:19	KRICHAR
Nickel (Ni)	142	ug/L		10	EPA 200.8	19-Oct-11 11:19	KRICHAR
Selenium (Se)	4110	ug/L		10	EPA 200.8	19-Oct-11 11:19	KRICHAR
Silver (Ag)	< 10	ug/L		10	EPA 200.8	19-Oct-11 11:19	KRICHAR
Zinc (Zn)	160	ug/L		20	EPA 200.8	19-Oct-11 11:19	KRICHAR

Site: BIOREACTOR 1 INF. Sample #: 2011021608

Collection Date: 12-Oct-11 10:29 AM Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	Method	Analysis Date/Time	Analyst			
TOTAL RECOVERABLE METALS B	Y ICP									
Boron (B)	152	mg/L		0.5	EPA 200.7	24-Oct-11 13:22	DJSULL1			
DISSOLVED METALS BY ICP-MS										
Selenium (Se)	1200	ug/L		10	EPA 200.8	19-Oct-11 12:07	KRICHAR			
TOTAL RECOVERABLE METALS BY ICP-MS										
Arsenic (As)	< 10	ug/L		10	EPA 200.8	19-Oct-11 11:22	KRICHAR			
Chromium (Cr)	11.9	ug/L		10	EPA 200.8	19-Oct-11 11:22	KRICHAR			
Copper (Cu)	< 10	ug/L		10	EPA 200.8	19-Oct-11 11:22	KRICHAR			
Nickel (Ni)	10.8	ug/L		10	EPA 200.8	19-Oct-11 11:22	KRICHAR			
Selenium (Se)	1240	ug/L		10	EPA 200.8	19-Oct-11 11:22	KRICHAR			
Silver (Ag)	< 10	ug/L		10	EPA 200.8	19-Oct-11 11:22	KRICHAR			
Zinc (Zn)	< 20	ug/L		20	EPA 200.8	19-Oct-11 11:22	KRICHAR			
SELENIUM SPECIATION										
Vendor Parameter	Complete				V_AS&C					

Site: BIOREACTOR 2 INF. Sample #: 2011021609

Collection Date: 12-Oct-11 10:35 AM Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	Method	Analysis Date/Time	Analyst
TOTAL RECOVERABLE METALS	BY ICP						
Boron (B)	154	mg/L		0.5	EPA 200.7	24-Oct-11 13:26	DJSULL1

This report shall not be reproduced, except in full.

Order # J11100039

Site: BIOREACTOR 2 INF.

Sample #:

2011021609

Collection Date: 12-Oct-11 10:35 AM

Matrix:

OTHER

Analyte	Result	Units	Qualifiers	RDL	Method	Analysis Date/Time	Analyst
TOTAL RECOVERABLE METALS	S BY ICP-MS						
Arsenic (As)	< 10	ug/L		10	EPA 200.8	19-Oct-11 11:25	KRICHAR
Chromium (Cr)	< 10	ug/L		10	EPA 200.8	19-Oct-11 11:25	KRICHAR
Copper (Cu)	< 10	ug/L		10	EPA 200.8	19-Oct-11 11:25	KRICHAR
Nickel (Ni)	15.9	ug/L		10	EPA 200.8	19-Oct-11 11:25	KRICHAR
Selenium (Se)	191	ug/L		10	EPA 200.8	19-Oct-11 11:25	KRICHAR
Silver (Ag)	< 10	ug/L		10	EPA 200.8	19-Oct-11 11:25	KRICHAR
Zinc (Zn)	< 20	ug/L		20	EPA 200.8	19-Oct-11 11:25	KRICHAR

Site: BIOREACTOR 2 EFF.

Sample #:

2011021610

Collection Date: 12-Oct-11 10:41 AM

Matrix:

OTHER

Analyte	Result	Units	Qualifiers	RDL	Method	Analysis Date/Time	Analyst				
INORGANIC IONS BY IC											
Bromide	78	mg/L		10	EPA 300.0	25-Oct-11 08:00	JAHERMA				
MERCURY (COLD VAPOR) IN WAT	ΓER										
Mercury (Hg)	< 1	ug/L		1	EPA 245.1	21-Oct-11 09:09	AGIBBS				
TOTAL RECOVERABLE METALS BY ICP											
Boron (B)	153	mg/L		0.5	EPA 200.7	24-Oct-11 13:30	DJSULL1				
TOTAL RECOVERABLE METALS BY ICP-MS											
Arsenic (As)	< 5	ug/L		5	EPA 200.8	19-Oct-11 11:28	KRICHAR				
Chromium (Cr)	< 5	ug/L		5	EPA 200.8	19-Oct-11 11:28	KRICHAR				
Copper (Cu)	< 5	ug/L		5	EPA 200.8	19-Oct-11 11:28	KRICHAR				
Nickel (Ni)	< 5	ug/L		5	EPA 200.8	19-Oct-11 11:28	KRICHAR				
Selenium (Se)	14.2	ug/L		5	EPA 200.8	19-Oct-11 11:28	KRICHAR				
Silver (Ag)	< 5	ug/L		5	EPA 200.8	19-Oct-11 11:28	KRICHAR				
Zinc (Zn)	< 10	ug/L		10	EPA 200.8	19-Oct-11 11:28	KRICHAR				
SELENIUM SPECIATION											
Vendor Parameter	Complete	•			V_AS&C						

Site: FILTER BLANK

Sample #:

2011021611

Collection Date: 12-Oct-11 10:22 AM

Matrix:

OTHER

Analyte	Result	Units	Qualifiers	RDL	Method	Analysis Date/Time	Analyst
DISSOLVED METALS BY ICP-MS							
Selenium (Se)	1.17	ug/L		1	EPA 200.8	19-Oct-11 10:42	KRICHAR

This report shall not be reproduced, except in full.

Order # J11100039

Site: Trip Blank Sample #: 2011021612 Collection Date: 12-Oct-11 10:22 AM Matrix: OTHER Analyte Result Units Qualifiers **RDL** Method **Analysis Date/Time** Analyst **TOTAL RECOVERABLE METALS BY ICP** Boron (B) 0.05 DJSULL1 < 0.05 mg/L EPA 200.7 24-Oct-11 12:59 **TOTAL RECOVERABLE METALS BY ICP-MS KRICHAR** Arsenic (As) < 1 ug/L 1 EPA 200.8 19-Oct-11 10:30 Chromium (Cr) < 1 ug/L 1 EPA 200.8 19-Oct-11 10:30 **KRICHAR** Copper (Cu) < 1 ug/L EPA 200.8 19-Oct-11 10:30 **KRICHAR** Nickel (Ni) ug/L 1 EPA 200.8 19-Oct-11 10:30 **KRICHAR** < 1 Selenium (Se) < 1 ug/L 1 **EPA 200.8** 19-Oct-11 10:30 **KRICHAR** Silver (Ag) < 1 ug/L EPA 200.8 19-Oct-11 10:30 **KRICHAR** < 2 ug/L 2 EPA 200.8 19-Oct-11 10:30 **KRICHAR** Zinc (Zn) **SELENIUM SPECIATION** Vendor Parameter Complete V_AS&C Site: BIOREACTOR 1 INF. Sample #: 2011021613 Collection Date: 12-Oct-11 10:29 AM Matrix: OTHER Qualifiers RDL Method Analyte Result Units Analysis Date/Time Analyst **MERCURY 1631** Vendor Parameter Complete V BRAND Site: HG BLANK BIOREACTOR 1 INF. Sample #: 2011021614 Collection Date: 12-Oct-11 10:29 AM Matrix: OTHER Analyte Result Units Qualifiers **RDL** Method **Analysis Date/Time Analyst MERCURY 1631**

Site: BIOREACTOR 2 INF. Sample #: 2011021615

Collection Date: 12-Oct-11 10:35 AM Matrix: OTHER

Analyte Result Units Qualifiers RDL Method Analysis Date/Time Analyst

V BRAND

MERCURY 1631

Vendor Parameter

Vendor Parameter Complete V_BRAND

Complete

This report shall not be reproduced, except in full.

Order # J11100039

Site: Hg Blk BioReactor 2 Inf Sample #: 2011021616

Collection Date: 12-Oct-11 10:35 AM Matrix: OTHER

Analyte Result Units Qualifiers RDL Method Analysis Date/Time Analyst

MERCURY 1631

Vendor Parameter Complete V_BRAND

Site: BIOREACTOR 2 EFF. Sample #: 2011021617

Collection Date: 12-Oct-11 10:41 AM Matrix: OTHER

Analyte Result Units Qualifiers RDL Method Analysis Date/Time Analyst

MERCURY 1631

Vendor Parameter Complete V_BRAND

Site: Hg Blk BioReactor 2 Eff Sample #: 2011021618

Collection Date: 12-Oct-11 10:41 AM Matrix: OTHER

Analyte Result Units Qualifiers RDL Method Analysis Date/Time Analyst

MERCURY 1631

Vendor Parameter Complete V_BRAND



18804 Northcreek Parkway Bothell, WA, 98011 Tel: (425) 483-3300 Fax: (425) 483-9818 www.appliedspeciation.com

October 21, 2011

Jay Perkins Duke Energy Analytical Laboratory Mail Code MGO3A2 (Building 7405) 13339 Hagers Ferry Rd. Huntersville, NC 28078 (704) 875-5245

Project: Belews - FGD WWTS (Bi-Monthly Sampling) (LIMS # J11100039)

Dear Mr. Perkins,

Attached is the report associated with four (4) aqueous samples submitted for selenium speciation analysis on October 13, 2011. The samples were received on October 14, 2011 in a sealed cooler at 0.7°C. Selenium speciation analysis was performed via ion chromatography inductively coupled plasma dynamic reaction cell mass spectrometry (IC-ICP-DRC-MS). Any issues associated with the analysis are addressed in the following report.

If you have any questions, please feel free to contact me at your convenience.

Sincerely,

Ben Wozniak Project Manager

Applied Speciation and Consulting, LLC

Ben Wozniek

Applied Speciation and Consulting, LLC

Report prepared for:

Jay Perkins
Duke Energy Analytical Laboratory
Mail Code MGO3A2 (Building 7405)
13339 Hagers Ferry Rd.
Huntersville, NC 28078

Project: Belews - FGD WWTS (Bi-Monthly Sampling) (LIMS # J11100039)

October 21, 2011

1. Sample Reception

Four (4) aqueous samples in 125mL HDPE bottles (provided by Applied Speciation and Consulting) were submitted for selenium speciation analysis on October 13, 2011. The samples were received on October 14, 2011 in a sealed container at 0.7°C.

The samples were received in a laminar flow clean hood void of trace metals contamination and ultra-violet radiation. Upon reception, the samples were designated discrete sample identifiers. An aliquot of each sample was filtered (0.45µm) and these filtrates were stored in a secure, monitored cryofreezer (maintained at a temperature of -80°C) until selenium speciation analysis could be performed via ion chromatography inductively coupled plasma dynamic reaction cell mass spectrometry (IC-ICP-DRC-MS).

2. Sample Preparation

All sample preparation is performed in laminar flow clean hoods known to be free from trace metals contamination. All applied water for dilutions and sample preservatives are monitored for contamination to account for any biases associated with the sample results.

<u>Selenium Speciation Analysis by IC-ICP-DRC-MS</u> Prior to analysis, an aliquot of each sample was filtered with a syringe filter (0.45µm) and injected directly into a sealed autosampler vial. No further sample preparation was performed as any chemical alteration of the samples may shift the equilibrium of the system resulting in changes in speciation ratios.

3. Sample Analysis

All sample analysis is preceded by a minimum of a five-point calibration curve spanning the entire concentration range of interest. Calibration curves are performed at the beginning of

each analytical day. All calibration curves, associated with each species of interest, are standardized by linear regression resulting in a response factor. All sample results are **instrument blank corrected** to account for any operational biases associated with the analytical platform.

Prior to sample analysis, all calibration curves are verified using second source standards which are identified as initial calibration verification standards (ICV).

Ongoing instrument performance is identified by the analysis of continuing calibration verification standards (CCV) and continuing calibration blanks (CCB) at a minimal interval of every ten analytical runs.

<u>Selenium Speciation Analysis by IC-ICP-DRC-MS</u> All samples for selenium speciation analysis were analyzed by ion chromatography inductively coupled plasma dynamic reaction cell mass spectrometry (IC-ICP-DRC-MS) on October 17-18, 2011. An aliquot of each sample is injected onto an anion exchange column and mobilized by a basic (pH > 7) gradient. The eluting selenium species are then introduced into a radio frequency (RF) plasma where energy-transfer processes cause desolvation, atomization, and ionization. The ions are extracted from the plasma through a differentially-pumped vacuum interface and travel through a pressurized chamber (DRC) containing a specific reactive gas which preferentially reacts with interfering ions of the same target mass to charge ratios (m/z). A solid-state detector detects ions transmitted through the mass analyzer and the resulting current is processed by a data handling system.

Retention times for each eluting species are compared to known standards for species identification.

4. Analytical Issues

The overall analyses went well and no significant analytical issues were encountered. All quality control parameters associated with these samples were within acceptance limits with the following exceptions:

The recoveries associated with the matrix spike (MS) and matrix spike duplicate (MSD) performed on the sample identified as Batch QC were below the established control limit of 75% for selenocyanate (54.4% and 51.2%, respectively). The MS and MSD also included selenite in the spiking solution which yielded elevated recoveries (139.1% and 143.3%, respectively). The low recoveries for selenocyanate correlate with the elevated recoveries of selenite suggesting that the sample matrix induces species conversion. The fact that no species conversion was observed in the ICV or CCVs, which contain both selenite and selenocyanate, demonstrates that the applied method stabilizes these selenium species in solution. Since the conversion of selenocyanate to selenite in the MS and MSD is a function of the sample matrix and the recoveries confirm a mass balance, no corrective action was required. The reported results are deemed representative of the supplied samples and suggest that selenocyanate is not stable in the spiked sample matrix.

The estimated method detection limits (eMDLs) for selenite, selenate, and selenocyanate are generated from replicate analyses of the lowest standard in the calibration curve. Not all selenium species are present in preparation blanks; therefore, eMDL calculations based on preparation blanks are artificially biased low.

The eMDL for methylseleninic acid and selenomethionine is calculated from the average eMDL of selenite, selenate, and selenocyanate. The calibration does not contain methylseleninic acid or selenomethionine due to impurities in these standards which would bias the results for other selenium species.

If you have any questions or concerns regarding this report, please feel free to contact me.

Sincerely,

Ben Wozniak

Project Manager

Applied Speciation and Consulting, LLC

Ben Woznick

Selenium Speciation Results for Duke Energy Project Name: Belews - FGD WWTS (Bi-Monthly Sampling) Contact: Jay Perkins LIMS #J11100039

Date: October 21, 2011
Report Generated by: Ben Wozniak
Applied Speciation and Consulting, LLC

Sample Results

						Unknown Se
Sample ID	Se(IV)	Se(VI)	SeCN	MeSe(IV)	SeMe	Species (n)
FGD Purge Eff	14.9	1030	ND (<1.3)	ND (<1.5)	ND (<1.5)	0 (0)
BioReactor 1 Inf	16.0	1070	ND (<0.34)	1.00	ND (<0.37)	0 (0)
BioReactor 2 Eff	1.61	6.40	ND (<0.34)	ND (<0.37)	ND (<0.37)	0 (0)
Metals Trip Blk	ND (<0.097)	ND (<0.055)	ND (<0.067)	ND (<0.073)	ND (<0.073)	0 (0)

All results reflect the applied dilution and are reported in µg/L

ND = Not detected at the applied dilution

SeCN = Selenocyanate

MeSe(IV) = Methylseleninic acid

SeMe = Selenomethionine

Unknown Se Species = Total concentration of all unknown Se species observed by IC-ICP-MS

n = number of unknown Se species observed

Selenium Speciation Results for Duke Energy Project Name: Belews - FGD WWTS (Bi-Monthly Sampling) Contact: Jay Perkins LIMS #J11100039

Date: October 21, 2011 Report Generated by: Ben Wozniak Applied Speciation and Consulting, LLC

Quality Control Summary - Preparation Blank Summary

Analyte (µg/L)	PBW1	PBW2	PBW3	PBW4	Mean	StdDev	eMDL*	eMDL 10x	eMDL 50x	eMDL 200x
Se(IV)	0.000	0.000	0.000	0.000	0.000	0.000	0.010	0.097	0.48	1.9
Se(VI)	0.000	0.000	0.000	0.000	0.000	0.000	0.006	0.055	0.28	1.1
SeCN	0.000	0.000	0.000	0.000	0.000	0.000	0.007	0.067	0.34	1.3
MeSe(IV)	0.000	0.000	0.000	0.000	0.000	0.000	0.007	0.073	0.37	1.5
SeMe	0.000	0.000	0.000	0.000	0.000	0.000	0.007	0.073	0.37	1.5

eMDL = Estimated Method Detection Limit

Quality Control Summary - Certified Reference Materials

Analyte (µg/L)	CRM	True Value	Result	Recovery
Se(IV)	LCS	9.57	11.24	117.5
Se(VI)	LCS	9.48	10.01	105.6
SeCN	LCS	8.92	9.239	103.6
MeSe(IV)	LCS	6.47	5.847	90.4
SeMe	LCS	9.32	9.200	98.7

^{*}Please see narrative regarding eMDL calculations

Selenium Speciation Results for Duke Energy Project Name: Belews - FGD WWTS (Bi-Monthly Sampling) Contact: Jay Perkins LIMS #J11100039

Date: October 21, 2011 Report Generated by: Ben Wozniak Applied Speciation and Consulting, LLC

Quality Control Summary - Matrix Duplicates

Analyte (µg/L)	Sample ID	Rep 1	Rep 2	Mean	RPD
Se(IV)	Batch QC*	12.0	11.9	11.9	8.0
Se(VI)	Batch QC*	973.3	1020	996.7	4.7
SeCN	Batch QC*	ND (<1.3)	ND (<1.3)	NC	NC
MeSe(IV)	Batch QC*	ND (<1.5)	ND (<1.5)	NC	NC
SeMe	Batch QC*	ND (<1.5)	ND (<1.5)	NC	NC

ND = Not detected at the applied dilution

NC = Value was not calculated due to one or more concentrations below the eMDL

Quality Control Summary - Matrix Spike/ Matrix Spike Duplicate

Analyte (µg/L)	Sample ID	Spike Conc	MS Result	Recovery	Spike Conc	MSD Result	Recovery	RPD
Se(IV)	Batch QC*	1112	1559	139.1**	1112	1606	143.3**	3.0
Se(VI)	Batch QC*	1009	2025	101.9	1009	2077	107.1	2.6
SeCN	Batch QC*	915.0	497.9	54.4**	915.0	468.2	51.2**	6.1

^{*} Batch QC performed on sample from LIMS # J11100235

^{*} Batch QC performed on sample from LIMS # J11100235

^{**} The recovery is outside the established control limits of 75-125%; please see narrative

CHAIN OF CUSTODY RECORD AND ANALYSIS REQUEST FORM **Duke Energy Analytical Laboratory** Analytical Laboratory Use Only Duke 16 of 27 age 1 of 2 ORDER# Mail Code MGO3A2 (Building 7405) **Energy**_s Originating J11100039 DISTRIBUTION 13339 Hagers Ferry Rd ORIGINAL to LAB Huntersville, N. C. 28078 (704) 875-5245 COPY to CLIENT SAMPLE PROGRAM Fax: (704) 875-4349 NPDES **Drinking Water** 1)Project Name AS&C Belews - FGD 2)Phone No: UST WWTS Bi-Monthly Sampling) RCRA Waste PO#133241 2) Client: 4)Fax No: 5Preserv.:1=HCL Bill Kennedy, Melonie Martin, 2=H2SO4 3=HNO3 Wayne Chapman, Tom Johnson ** 4=Ice 5=None 4 3.4 4 3.4 5)Business Unit: 6)Process: MR # 16 Analyses Required Mail Code: dig.) 8)Oper. Unit: 9)Res. Type: 10)Reso. Center: Customer to complete all ou) appropriate non-shaded areas. Br (Dionex) soluble 245.1 Sampling conducted: 2nd and 4th Wednesday LAB USE ONLY Metals* Se Speciation Bottle TDS Hg-Se, ¹³Sample Description or ID Date Time Signature FGD Purge Eff 70 EQ Tank Eff. 10:56 70 BioReactor 1 Inf BioReactor 2 Inf BioReactor 2 Eff 1 1 10/12 10:22 Filter Blk 1 Metals Trip Blk Filtering of the Se is performed in the field please provide a filter blank too. 1) Relinquished B ²²Requested Turnaround 3) Retinguished By 14 Days IMPORTANT 6)Accepted By 8)Accepted By: Date/Time 9)Seal/Locked By * Add. Cost Will Apply 11)Seal/Locked By Comments: * B by ICP As, Cr, Cu, Ni, Se, Ag, Zn by IMS Digestions = TRM thomas.d.johnson@siemens.com



October 26, 2011

Duke Energy
ATTN: Jay Perkins
Scientific Support-Laboratory
13339 Hagers Ferry Road
Huntersville NC 28078
jcperkins@duke-energy.com
labcustomer@duke-energy.com

RE: Project DUK-HV1101 Client Project: J11100039

Dear Mr. Perkins,

On October 14, 2011, Brooks Rand Labs (BRL) received three (3) flue gas desulfurization (FGD) wastewater samples and three (3) corresponding blank samples. Samples were logged-in for total mercury (Hg) analysis. All samples were received, prepared, analyzed, and stored according to BRL SOPs and EPA methodology.

The results were blank-corrected as described in the calculations section of the applicable SOP(s) and may be evaluated using adjusted reporting limits to account for sample aliquot size. Please refer to the *Sample Results* page for sample-specific detection limits and other details. Aside from concentration qualifiers, all data was reported without qualification and all associated quality control sample results met the acceptance criteria.

It should be noted that a matrix spike/matrix spike duplicate (MS/MSD) set was not analyzed on one of the samples from this work order. A similar sample from a different work order had a MS/MSD set analyzed and that has been reported.

The sample *Hg Blk BioReactor 2 Eff* (1142048-06) was identified as a field blank, but had a detectable result that was comparable to the associated sample. It was also noticed that when preserved, *Hg Blk BioReactor 2 Eff* (1142048-06) reacted similarly to a sample and not a field blank.

BRL, an accredited laboratory, certifies the reported results of all analyses for which BRL is NELAP accredited meet all NELAP requirements. For more details, see the *Report Information* page of the report.

Please feel free to contact me if you have any questions regarding this report.

Sincerely,

Lydia Greaves
Project Manager
lydia@brooksran.com

Project ID: DUK-HV1101 **PM:** Tiffany Stilwater



Page 18 of 27 Client PM: Jay Perkins Client PO: 141391

Report Information

Laboratory Accreditation

BRL is accredited by the *National Environmental Laboratory Accreditation Program* (NELAP) through the State of Florida Department of Health, Bureau of Laboratories (E87982) and is certified to perform many environmental analyses. BRL is also certified by many other states to perform environmental analyses. For a current list of our accreditations/certifications, please visit our website at http://www.brooksrand.com/default.asp?contentID=586. Results reported relate only to the samples listed in the report.

Field Quality Control Samples

Please be notified that certain EPA methods require the collection of field quality control samples of an appropriate type and frequency; failure to do so is considered a deviation from some methods and for compliance purposes should only be done with the approval of regulatory authorities. Please see the specific EPA methods for details regarding required field quality control samples.

Common Abbreviations

BLK	method blank	MS	matrix spike
BRL	Brooks Rand Labs	MSD	matrix spike duplicate
BS	laboratory fortified blank	ND	non-detect
CAL	calibration standard	NR	non-reportable
CCV	continuing calibration verification	PS	post preparation spike
COC	chain of custody record	REC	percent recovery
CRM	certified reference material	RPD	relative percent difference
D	dissolved fraction	RSD	relative standard deviation
DUP	duplicate	SCV	secondary calibration verification
ICV	initial calibration verification	SOP	standard operating procedure
MDL	method detection limit	SRM	standard reference material
MRL	method reporting limit	T	total recoverable fraction

Definition of Data Qualifiers

(Effective 9/23/09)

- B Detected by the instrument, the result is > the MDL but ≤ the MRL. Result is reported and considered an estimate.
- **E** An estimated value due to the presence of interferences. A full explanation is presented in the narrative.
- **H** Holding time and/or preservation requirements not met. Result is estimated.
- **J** Estimated value. A full explanation is presented in the narrative.
- J-M Duplicate precision (RPD) for associated QC sample was not within acceptance criteria. Result is estimated.
- J-N Spike recovery for associated QC sample was not within acceptance criteria. Result is estimated.
- M Duplicate precision (RPD) was not within acceptance criteria. Result is estimated.
- N Spike recovery was not within acceptance criteria. Result is estimated.
- **R** Rejected, unusable value. A full explanation is presented in the narrative.
- U Result is ≤ the MDL or client requested reporting limit (CRRL). Result reported as the MDL or CRRL.
- X Result is not BLK-corrected and is within 10x the absolute value of the highest detectable BLK in the batch. Result is estimated.

These qualifiers are based on those previously utilized by Brooks Rand, Ltd., those found in the EPA <u>SOW ILM03.0</u>, Exhibit B, Section III, pg. B-18, and the <u>USEPA Laboratory Data Validation Functional Guidelines for Evaluating Inorganic Analyses; USEPA; July 2002. These supersede all previous qualifiers ever employed by BRL.</u>

Project ID: DUK-HV1101 **PM:** Tiffany Stilwater



Page 19 of 27 Client PM: Jay Perkins Client PO: 141391

Sample Information

Sample	Lab ID	Report Matrix	Type	Sampled	Received
BioReactor 1 Inf	1142048-01	Influent	Sample	10/12/2011	10/14/2011
Hg Blk BioReactor 1 Inf	1142048-02	DIW	Field Blank	10/12/2011	10/14/2011
BioReactor 2 Inf	1142048-03	Influent	Sample	10/12/2011	10/14/2011
Hg Blk BioReactor 2 Inf	1142048-04	DIW	Field Blank	10/12/2011	10/14/2011
BioReactor 2 Eff	1142048-05	Effluent	Sample	10/12/2011	10/14/2011
Hg Blk Bio Reactor 2 Eff	1142048-06	DIW	Field Blank	10/12/2011	10/14/2011

Batch Summary

Analyte	Lab Matrix	Method	Prepared	Analyzed	Batch	Sequence
Hg	Water	EPA 1631	10/21/2011	10/25/2011	B111723	1100738



Page 20 of 27 Client PM: Jay Perkins Client PO: 141391

Sample Results

Sample	Analyte	Report Matrix	Fraction	Result	Qualifier	MDL	MRL	Unit	Batch	Sequence
BioReactor 1 In 1142048-01	f Hg	Influent	Т	41700		76.5	204	ng/L	B111723	1100738
BioReactor 2 E 1142048-05	ff Hg	Effluent	Т	412		3.03	8.08	ng/L	B111723	1100738
BioReactor 2 In 1142048-03	f Hg	Influent	Т	2460		15.3	40.8	ng/L	B111723	1100738
Hg Blk Bio Read 1142048-06	ctor 2 Eff Hg	DIW	Т	527		1.52	4.04	ng/L	B111723	1100738
Hg Blk BioRead 1142048-02	e tor 1 Inf Hg	DIW	Т	0.38	В	0.15	0.41	ng/L	B111723	1100738
Hg Blk BioRead 1142048-04	c tor 2 Inf Hg	DIW	Т	0.15	U	0.15	0.41	ng/L	B111723	1100738



Page 21 of 27 Client PM: Jay Perkins Client PO: 141391

Accuracy & Precision Summary

Batch: B111723 Lab Matrix: Water Method: EPA 1631

Sample B111723-SRM1	Analyte Certified Reference Materia	Native al (1140052	Spike	Result	Units	REC & Limits	RPD & Limits
	Hg	`	15.68	14.44	ng/L	92% 85-115	
B111723-MS2	Matrix Spike (1143014-01) Hg	436.0	2020	2506	ng/L	102% 71-125	
B111723-MSD2	Matrix Spike Duplicate (114 Hg	13014-01) 436.0	2020	2473	ng/L	101% 71-125	1% 24

Method Blanks & Reporting Limits

Batch: B111723 Matrix: Water Method: EPA 1631 Analyte: Hg

Sample	Result	Units
B111723-BLK1	0.04	ng/L
B111723-BLK2	0.0008	ng/L
B111723-BLK3	0.05	ng/L
B111723-BLK4	0.02	na/L

 Average: 0.03
 Standard Deviation: 0.02
 MDL: 0.15

 Limit: 0.50
 Limit: 0.10
 MRL: 0.41

Project ID: DUK-HV1101 PM: Tiffany Stilwater



Page 22 of 27 Client PM: Jay Perkins **Client PO: 141391**

Instrument Calibration

Sequence: 1100738 **Total Mercury and Mercury Speciation by CVAFS** Instrument: THG-05

Method: EPA 1631

Date: 10/25/2011 Analyte: Hg

Lab ID	True Value	Result	Units	REC	C & Limits
1100738-IBL1		8.42	pg of Hg		
1100738-IBL2		8.90	pg of Hg		
1100738-IBL3		7.23	pg of Hg		
1100738-IBL4		8.50	pg of Hg		
1100738-CAL1	25.00	24.03	pg of Hg	96%	
1100738-CAL2	100.0	99.48	pg of Hg	99%	
1100738-CAL3	500.0	511.0	pg of Hg	102%	
1100738-CAL4	2500	2549	pg of Hg	102%	
1100738-CAL5	10000	10050	pg of Hg	100%	
1100738-ICV1	1568	1444	pg of Hg	92%	85-115
1100738-CCB1		12.1	pg of Hg		
1100738-CCV1	500.0	514.6	pg of Hg	103%	77-123
1100738-CCB2		8.55	pg of Hg		
1100738-CCV2	500.0	497.9	pg of Hg	100%	77-123
1100738-CCB3		37.1	pg of Hg		
1100738-CCV3	500.0	512.1	pg of Hg	102%	77-123

Project ID: DUK-HV1101 **PM:** Tiffany Stilwater



Page 23 of 27 Client PM: Jay Perkins Client PO: 141391

Sample Containers

	ID: 1142048-01 ple: BioReactor 1 Inf		-	rt Matrix: Influent le Type: Sample		Collected: 10/12/2011 Received: 10/14/2011		
Des A	Container Bottle FLPE Hg-T	Size 250mL	Lot 71443390 30	Preservation none	P-Lot n/a	рН	Ship. Cont. Cooler	
В	EXTRA_VOL	250 mL	71443390 30	none	n/a		Cooler	
	ID: 1142048-02 ple: Hg Blk BioReactor 1 Inf		-	rt Matrix: DIW le Type: Field Blank			cted: 10/12/2011 ived: 10/14/2011	
Des A	Container Bottle FLPE Hg-T	Size 250mL	Lot 71443390 30	Preservation none	P-Lot n/a	рН	Ship. Cont. Cooler	
В	EXTRA_VOL	250 mL	71443390 30	none	n/a		Cooler	
	Lab ID: 1142048-03 Sample: BioReactor 2 Inf		•	rt Matrix: Influent le Type: Sample			cted: 10/12/2011 ived: 10/14/2011	
Des A	Container Bottle FLPE Hg-T	Size 250mL	Lot 71443390 30	Preservation none	P-Lot n/a	рН	Ship. Cont. Cooler	
В	EXTRA_VOL	250 mL	71443390 30	none	n/a		Cooler	
	ID: 1142048-04 ple: Hg Blk BioReactor 2 Inf		Repo Samp			cted: 10/12/2011 ived: 10/14/2011		
Des A	Container Bottle FLPE Hg-T	Size 250mL	Lot 71443390 30	Preservation none	P-Lot n/a	рН	Ship. Cont. Cooler	
В	EXTRA_VOL	250 mL	71443390 30	none	n/a		Cooler	
	ID: 1142048-05 ple: BioReactor 2 Eff		Report Matrix: Effluent Sample Type: Sample				cted: 10/12/2011 ived: 10/14/2011	
Des A	Container Bottle FLPE Hg-T	Size 250mL	Lot 71443390 30	Preservation none	P-Lot n/a	рН	Ship. Cont. Cooler	
В	EXTRA_VOL	250 mL	71443390 30	none	n/a		Cooler	

Project ID: DUK-HV1101 **PM:** Tiffany Stilwater



Page 24 of 27 Client PM: Jay Perkins Client PO: 141391

Collected: 10/12/2011

Received: 10/14/2011

Sample Containers

Lab ID: 1142048-06Report Matrix: DIWSample: Hg Blk Bio Reactor 2 EffSample Type: Field Blank

DesContainerSizeLotPreservationP-LotpHShip. Cont.ABottle FLPE Hg-T250mL71443390nonen/aCooler30

none

EXTRA_VOL 250 mL 71443390

30

n/a Cooler

Shipping Containers

Cooler

В

Received: October 14, 2011 9:00
Tracking No: 472679664810 via FedEx

Coolant Type: Ice Temperature: 3.6 °C Description: Cooler
Damaged in transit? No
Returned to client? No

Custody seals present? No Custody seals intact? No COC present? Yes

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